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2-{[2-(1-Methyl-2,2-dioxo-3,4-dihydro-1*H*-2λ^6,1-benzothiazin-4-ylidene)-hydrazin-1-ylidene]methyl}phenol

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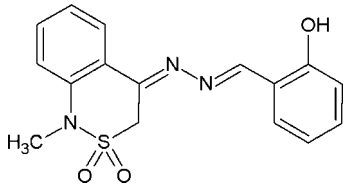
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.114; data-to-parameter ratio = 17.8.

In the title compound, $C_{16}H_{15}N_3O_3S$, the dihedral angle between the aromatic rings is 8.18 (11)° and the C=N—N=C torsion angle is 178.59 (14)°. The conformation of the thiazine ring is an envelope, with the S atom displaced by 0.8157 (18) Å from the mean plane of the other five atoms (r.m.s. deviation = 0.045 Å). An intramolecular O—H···N hydrogen bond closes an S(6) ring. In the crystal, weak C—H···O interactions link the molecules, with all three O atoms acting as acceptors.

Related literature

For the synthesis and biological activity of the title compound and related materials, see: Shafiq *et al.* (2011).



Experimental

Crystal data

$C_{16}H_{15}N_3O_3S$
M_r = 329.37
Monoclinic, $P2_1/n$
a = 6.5530 (2) Å
b = 15.8719 (5) Å
c = 14.5804 (4) Å
β = 91.147 (1)°
V = 1516.18 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 296 K
0.40 × 0.05 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer
14391 measured reflections
3778 independent reflections
3019 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.114
S = 1.03
3778 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···N3	0.85 (2)	1.89 (2)	2.6574 (18)	151 (2)
C1—H1B···O3^{i}	0.96	2.59	3.534 (3)	166
C3—H3A···O1^{ii}	0.93	2.60	3.396 (2)	144
C9—H9A···O2^{i}	0.97	2.44	3.138 (2)	129
C16—H16···O1^{iii}	0.93	2.60	3.440 (2)	151

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x+1, y, z$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *ORTEP-3* (Farrugia, 1997).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5989).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

Acta Cryst. (2012). E**68**, o2643 [doi:10.1107/S1600536812034101]

2-{[2-(1-Methyl-2,2-dioxo-3,4-dihydro-1*H*-2λ^6,1-benzothiazin-4-ylidene)hydrazin-1-ylidene]methyl}phenol

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Experimental

In the synthesis of title compound, 4-hydrazinylidene-1- methyl-3H-2λ^6,1-benzothiazine-2,2-dione was subjected to react with 2-hydroxy benzaldehyde according to literature procedure ((Shafiq *et al.* (2011)). The product obtained was then recrystallized in ethylacetate under slow evaporation to obtain single crystals suitable for X-ray diffraction.

Refinement

The O-bond H atom was located in a difference map and its position was freely refined. The C-bound H-atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding. The methyl group was allowed to rotate, but not to tip, to best fit the electron density. The constraint U_{iso}(H) = 1.2U_{eq}(C,O) or 1.5U_{eq}(methyl C) was applied.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *ORTEP-3* (Farrugia, 1997).

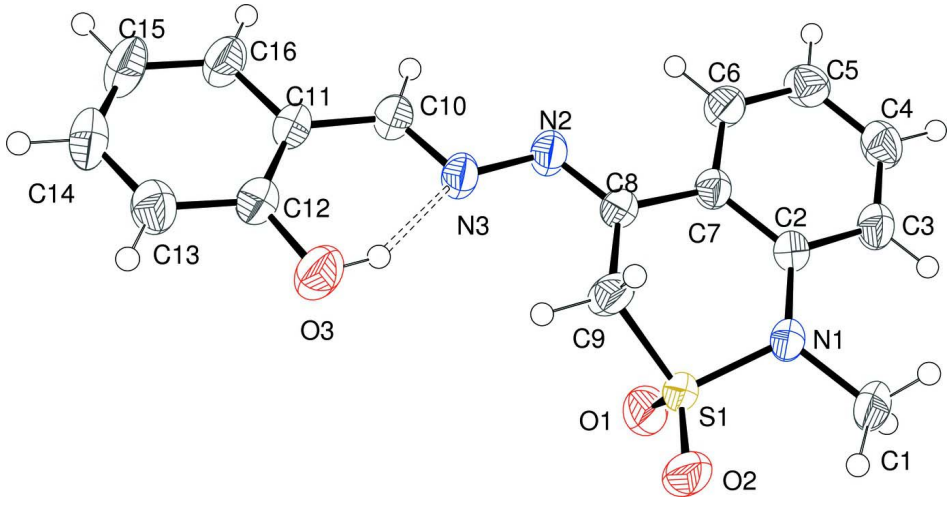

Figure 1

The molecular structure of (I), showing displacement ellipsoids at the 50% probability level. The hydrogen bond is shown as a double-dashed line.

2-{[2-(1-Methyl-2,2-dioxo-3,4-dihydro-1*H*-2λ^6,1-benzothiazin- 4-ylidene)hydrazin-1-ylidene]methyl}phenol

Crystal data

$C_{16}H_{15}N_3O_3S$
M_r = 329.37
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 6.5530 (2) Å
b = 15.8719 (5) Å
c = 14.5804 (4) Å
β = 91.147 (1)°
V = 1516.18 (8) Å^3
Z = 4
$F(000)$ = 688
D_x = 1.443 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6831 reflections
θ = 2.8–28.2°
μ = 0.23 mm^{-1}
T = 296 K
Needle, yellow
0.40 × 0.05 × 0.05 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
14391 measured reflections
3778 independent reflections
3019 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 28.4°, θ_{min} = 2.6°
h = −8→8
k = −21→20
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.114
S = 1.03
3778 reflections
212 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.5143P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.5578 (3)	0.63906 (15)	0.45564 (14)	0.0583 (5)
H1A	0.5592	0.6923	0.4245	0.087*
H1B	0.5077	0.5963	0.4144	0.087*
H1C	0.6939	0.6250	0.4760	0.087*
C2	0.4661 (2)	0.70248 (9)	0.60704 (10)	0.0323 (3)

C3	0.6391 (2)	0.75336 (11)	0.60255 (12)	0.0418 (4)
H3A	0.7228	0.7498	0.5519	0.050*
C4	0.6875 (3)	0.80886 (11)	0.67218 (12)	0.0453 (4)
H4	0.8039	0.8420	0.6683	0.054*
C5	0.5651 (3)	0.81578 (11)	0.74765 (12)	0.0436 (4)
H5	0.5973	0.8538	0.7942	0.052*
C6	0.3944 (2)	0.76554 (10)	0.75302 (11)	0.0384 (3)
H6	0.3127	0.7699	0.8042	0.046*
C7	0.3401 (2)	0.70828 (9)	0.68407 (10)	0.0318 (3)
C8	0.1578 (2)	0.65501 (9)	0.69491 (10)	0.0323 (3)
C9	0.1241 (3)	0.58446 (12)	0.62738 (11)	0.0470 (4)
H9A	−0.0177	0.5670	0.6280	0.056*
H9B	0.2080	0.5366	0.6450	0.056*
C10	−0.2398 (2)	0.63187 (10)	0.83713 (11)	0.0375 (3)
H10	−0.2025	0.6761	0.8758	0.045*
C11	−0.4218 (2)	0.58389 (10)	0.85695 (10)	0.0357 (3)
C12	−0.4875 (2)	0.51625 (10)	0.80224 (11)	0.0383 (3)
C13	−0.6624 (3)	0.47198 (12)	0.82474 (13)	0.0490 (4)
H13	−0.7045	0.4263	0.7891	0.059*
C14	−0.7732 (3)	0.49541 (13)	0.89940 (15)	0.0566 (5)
H14	−0.8905	0.4657	0.9138	0.068*
C15	−0.7125 (3)	0.56253 (13)	0.95334 (14)	0.0584 (5)
H15	−0.7889	0.5782	1.0036	0.070*
C16	−0.5388 (3)	0.60604 (12)	0.93243 (12)	0.0474 (4)
H16	−0.4979	0.6511	0.9691	0.057*
S1	0.18651 (6)	0.61737 (3)	0.51673 (2)	0.03890 (13)
N1	0.42477 (18)	0.64443 (9)	0.53490 (9)	0.0382 (3)
N2	0.03875 (19)	0.67010 (8)	0.76209 (9)	0.0378 (3)
N3	−0.12812 (19)	0.61537 (8)	0.76789 (9)	0.0374 (3)
O1	0.06395 (19)	0.68861 (10)	0.49308 (9)	0.0581 (4)
O2	0.1846 (2)	0.54798 (10)	0.45479 (9)	0.0610 (4)
O3	−0.3885 (2)	0.49217 (9)	0.72635 (9)	0.0557 (4)
H3	−0.285 (4)	0.5240 (15)	0.7235 (16)	0.067*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0359 (8)	0.0874 (14)	0.0524 (11)	−0.0090 (9)	0.0175 (7)	−0.0246 (10)
C2	0.0275 (6)	0.0353 (7)	0.0342 (7)	−0.0008 (5)	0.0019 (5)	0.0000 (6)
C3	0.0329 (7)	0.0478 (9)	0.0448 (9)	−0.0081 (6)	0.0076 (6)	−0.0008 (7)
C4	0.0379 (8)	0.0441 (9)	0.0537 (10)	−0.0121 (7)	0.0005 (7)	0.0002 (8)
C5	0.0475 (9)	0.0395 (8)	0.0437 (9)	−0.0083 (7)	−0.0031 (7)	−0.0046 (7)
C6	0.0411 (8)	0.0380 (8)	0.0363 (8)	−0.0042 (6)	0.0048 (6)	−0.0022 (6)
C7	0.0294 (6)	0.0327 (7)	0.0335 (7)	−0.0012 (5)	0.0025 (5)	0.0028 (6)
C8	0.0323 (7)	0.0351 (7)	0.0297 (7)	−0.0033 (6)	0.0024 (5)	0.0026 (6)
C9	0.0585 (10)	0.0508 (10)	0.0323 (8)	−0.0227 (8)	0.0126 (7)	−0.0050 (7)
C10	0.0363 (7)	0.0381 (8)	0.0383 (8)	−0.0018 (6)	0.0072 (6)	0.0000 (6)
C11	0.0341 (7)	0.0371 (8)	0.0362 (7)	0.0011 (6)	0.0087 (6)	0.0045 (6)
C12	0.0389 (8)	0.0383 (8)	0.0381 (8)	0.0004 (6)	0.0059 (6)	0.0039 (6)
C13	0.0463 (9)	0.0464 (9)	0.0543 (10)	−0.0108 (8)	0.0046 (8)	0.0018 (8)

C14	0.0418 (9)	0.0618 (12)	0.0668 (12)	−0.0125 (8)	0.0164 (8)	0.0091 (10)
C15	0.0516 (10)	0.0650 (12)	0.0598 (11)	−0.0021 (9)	0.0295 (9)	0.0004 (10)
C16	0.0485 (9)	0.0491 (10)	0.0453 (9)	−0.0028 (8)	0.0171 (7)	−0.0049 (7)
S1	0.03155 (19)	0.0558 (3)	0.0296 (2)	−0.01232 (16)	0.00564 (13)	−0.00418 (16)
N1	0.0279 (6)	0.0499 (8)	0.0371 (7)	−0.0062 (5)	0.0078 (5)	−0.0085 (6)
N2	0.0333 (6)	0.0401 (7)	0.0403 (7)	−0.0054 (5)	0.0083 (5)	−0.0013 (5)
N3	0.0331 (6)	0.0396 (7)	0.0397 (7)	−0.0053 (5)	0.0084 (5)	0.0011 (5)
O1	0.0377 (6)	0.0885 (10)	0.0479 (7)	0.0080 (6)	−0.0021 (5)	0.0094 (7)
O2	0.0638 (8)	0.0775 (9)	0.0423 (7)	−0.0335 (7)	0.0167 (6)	−0.0228 (6)
O3	0.0623 (8)	0.0551 (8)	0.0505 (7)	−0.0147 (6)	0.0214 (6)	−0.0147 (6)

Geometric parameters (Å, º)

C1—N1	1.4641 (19)	C9—H9B	0.9700
C1—H1A	0.9600	C10—N3	1.286 (2)
C1—H1B	0.9600	C10—C11	1.449 (2)
C1—H1C	0.9600	C10—H10	0.9300
C2—C3	1.394 (2)	C11—C16	1.399 (2)
C2—C7	1.4104 (19)	C11—C12	1.400 (2)
C2—N1	1.4203 (19)	C12—O3	1.349 (2)
C3—C4	1.376 (2)	C12—C13	1.389 (2)
C3—H3A	0.9300	C13—C14	1.372 (3)
C4—C5	1.379 (2)	C13—H13	0.9300
C4—H4	0.9300	C14—C15	1.378 (3)
C5—C6	1.377 (2)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.371 (3)
C6—C7	1.396 (2)	C15—H15	0.9300
C6—H6	0.9300	C16—H16	0.9300
C7—C8	1.4745 (19)	S1—O2	1.4242 (13)
C8—N2	1.2870 (19)	S1—O1	1.4252 (15)
C8—C9	1.504 (2)	S1—N1	1.6358 (13)
C9—S1	1.7519 (16)	N2—N3	1.4004 (17)
C9—H9A	0.9700	O3—H3	0.85 (2)
N1—C1—H1A	109.5	N3—C10—C11	122.22 (15)
N1—C1—H1B	109.5	N3—C10—H10	118.9
H1A—C1—H1B	109.5	C11—C10—H10	118.9
N1—C1—H1C	109.5	C16—C11—C12	118.29 (14)
H1A—C1—H1C	109.5	C16—C11—C10	119.41 (15)
H1B—C1—H1C	109.5	C12—C11—C10	122.30 (14)
C3—C2—C7	119.28 (14)	O3—C12—C13	117.56 (16)
C3—C2—N1	118.96 (13)	O3—C12—C11	122.50 (14)
C7—C2—N1	121.73 (13)	C13—C12—C11	119.93 (15)
C4—C3—C2	120.75 (15)	C14—C13—C12	120.17 (17)
C4—C3—H3A	119.6	C14—C13—H13	119.9
C2—C3—H3A	119.6	C12—C13—H13	119.9
C3—C4—C5	120.71 (15)	C13—C14—C15	120.74 (16)
C3—C4—H4	119.6	C13—C14—H14	119.6
C5—C4—H4	119.6	C15—C14—H14	119.6
C6—C5—C4	119.02 (15)	C16—C15—C14	119.59 (17)

C6—C5—H5	120.5	C16—C15—H15	120.2
C4—C5—H5	120.5	C14—C15—H15	120.2
C5—C6—C7	122.12 (15)	C15—C16—C11	121.26 (17)
C5—C6—H6	118.9	C15—C16—H16	119.4
C7—C6—H6	118.9	C11—C16—H16	119.4
C6—C7—C2	118.11 (13)	O2—S1—O1	117.53 (9)
C6—C7—C8	119.44 (13)	O2—S1—N1	107.57 (8)
C2—C7—C8	122.44 (13)	O1—S1—N1	111.25 (8)
N2—C8—C7	118.66 (13)	O2—S1—C9	110.74 (8)
N2—C8—C9	123.59 (13)	O1—S1—C9	108.58 (9)
C7—C8—C9	117.74 (13)	N1—S1—C9	99.72 (8)
C8—C9—S1	110.29 (11)	C2—N1—C1	120.96 (13)
C8—C9—H9A	109.6	C2—N1—S1	117.24 (10)
S1—C9—H9A	109.6	C1—N1—S1	116.01 (11)
C8—C9—H9B	109.6	C8—N2—N3	114.62 (13)
S1—C9—H9B	109.6	C10—N3—N2	112.24 (13)
H9A—C9—H9B	108.1	C12—O3—H3	105.7 (16)
C7—C2—C3—C4	0.0 (2)	C11—C12—C13—C14	1.3 (3)
N1—C2—C3—C4	−178.31 (15)	C12—C13—C14—C15	−0.4 (3)
C2—C3—C4—C5	−0.5 (3)	C13—C14—C15—C16	−0.4 (3)
C3—C4—C5—C6	0.8 (3)	C14—C15—C16—C11	0.3 (3)
C4—C5—C6—C7	−0.6 (3)	C12—C11—C16—C15	0.6 (3)
C5—C6—C7—C2	0.1 (2)	C10—C11—C16—C15	179.69 (18)
C5—C6—C7—C8	178.70 (15)	C8—C9—S1—O2	171.30 (12)
C3—C2—C7—C6	0.2 (2)	C8—C9—S1—O1	−58.26 (15)
N1—C2—C7—C6	178.42 (14)	C8—C9—S1—N1	58.19 (14)
C3—C2—C7—C8	−178.33 (14)	C3—C2—N1—C1	−1.5 (2)
N1—C2—C7—C8	−0.1 (2)	C7—C2—N1—C1	−179.75 (16)
C6—C7—C8—N2	8.8 (2)	C3—C2—N1—S1	−153.63 (13)
C2—C7—C8—N2	−172.73 (14)	C7—C2—N1—S1	28.14 (19)
C6—C7—C8—C9	−169.98 (15)	O2—S1—N1—C2	−169.05 (12)
C2—C7—C8—C9	8.5 (2)	O1—S1—N1—C2	60.90 (13)
N2—C8—C9—S1	141.30 (14)	C9—S1—N1—C2	−53.51 (14)
C7—C8—C9—S1	−40.03 (19)	O2—S1—N1—C1	37.45 (16)
N3—C10—C11—C16	−178.83 (16)	O1—S1—N1—C1	−92.59 (15)
N3—C10—C11—C12	0.2 (2)	C9—S1—N1—C1	152.99 (15)
C16—C11—C12—O3	177.69 (16)	C7—C8—N2—N3	−178.86 (12)
C10—C11—C12—O3	−1.4 (2)	C9—C8—N2—N3	−0.2 (2)
C16—C11—C12—C13	−1.4 (2)	C11—C10—N3—N2	179.66 (14)
C10—C11—C12—C13	179.55 (15)	C8—N2—N3—C10	178.59 (14)
O3—C12—C13—C14	−177.81 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···N3	0.85 (2)	1.89 (2)	2.6574 (18)	151 (2)
C1—H1*B*···O3^{i}	0.96	2.59	3.534 (3)	166
C3—H3*A*···O1^{ii}	0.93	2.60	3.396 (2)	144

C9—H9*A*···O2^{i}	0.97	2.44	3.138 (2)	129
C16—H16···O1^{iii}	0.93	2.60	3.440 (2)	151

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*+1, *y*, *z*; (iii) *x*−1/2, −*y*+3/2, *z*+1/2.